

TECHNICAL REPORT NATICK/TR-02/007

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# EXPERIMENTAL BATTLEDRESS UNIFORM FABRICS MADE FROM AMINE OXIDE SOLVENT SPUN CELLULOSIC FIBERS

by C. Winterhalter

February 2002

Final Report October 1992 - August 1995

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#### PREFACE

Courtaulds Fiber, Limited, of the United Kingdom has developed a new type of rayon called lyocell and uses the trade name "Tencel" in the United States. The fiber has been advertised as being stronger and softer than standard rayon fiber. The Natick Soldier Center established an effort to investigate this new fiber and determine if it would provide any performance improvements over existing materials used in combat uniform fabrics. In a leveraged effort with Courtaulds, Natick developed, tested and evaluated several Tencel based fabrics that were camouflage printed and either durable press treated or flame resistant treated. Overall the treated fabrics did not provide any improvements over existing combat uniform fabrics.

This project was undertaken during the period October 1992 to August 1995, and funded under the Program Element Number 622786 and Project Number AH98.

# EXPERIMENTAL BATTLEDRESS UNIFORM FABRICS MADE FROM AMINE OXIDE SOLVENT SPUN CELLULOSIC FIBERS

## **SUMMARY**

The potential of an amine oxide solvent spun cellulosic fiber called Tencel<sup>®</sup>, was explored for use as a battledress uniform fabric. An experimental 100 percent Tencel fabric was woven and printed with a woodland camouflage pattern. Half was finished with a durable press treatment (DPT) and the remainder with a flame-retardant treatment (FRT).

The Tencel fabrics were evaluated and compared to the former all-cotton standard Hot Weather Battledress Uniform fabric and the current standard 50 percent nylon, 50 percent cotton Hot Weather Battledress Uniform fabric. The Tencel demonstrated physical properties that were approximately equal or superior to the other fabrics in breaking strength, tearing strength and air permeability. Its abrasion resistance was lower than the nylon/cotton fabric. Colorfastness, visual shade, near infrared spectral reflectance, and appearance were not acceptable. The FRT Tencel fabric demonstrated increased fabric weight and relatively low breaking and tearing strengths. Both the DPT and FRT Tencel fabrics are not recommended to replace the standard military combat uniform fabric.

In a follow-on effort, 50 percent Type 420 nylon and 50 percent Tencel yarn blends were successfully spun using commercial equipment. Substitution of Tencel for cotton in nylon/cotton ripstop poplin fabrics is recommended for further development and evaluation.

#### INTRODUCTION

The manufacture of rayon has historically been known to be an environmentally unfriendly Two European fiber manufacturers, Courtaulds and Lenzing AG, Austria, developed an environmentally friendly manufacturing process where the fibers are solvent spun. Wood pulp and amine oxide are mixed, and passed through a continuous dissolving unit that yields a clear, very viscous solution. It is then spun in a dilute aqueous solution of the amine oxide that precipitates the cellulose as continuous filaments. The fibers are washed and dried in tow form, cut into staple and baled. The amine oxide is recovered, thereby eliminating environmental problems.<sup>1,2</sup> Fibers made from this process are known as lyocell. Approval from the U.S. Federal Trade Commission (FTC) to use the European generic designation, lyocell, has been granted. Both rayon and lyocell are made from regenerated cellulose. However, according to the FTC's "Rules and Regulations Under the Textile Fiber Products Identification Act," dated January 1999, while lyocell is listed as a subset of rayon, it differs from rayon when the cellulose is precipitated from an organic solution in which no substitution of the hydroxyl groups takes place and no chemical intermediates are formed. Rayon is defined as regenerated cellulose in which constituents have replaced not more than 15 percent of the hydrogens of the hydroxyl groups.

Tencel is the U.S. trademark for Courtaulds lyocell fiber. Tencel is marketed and manufactured in the U.S. by its wholly owned American subsidiary, Courtaulds Fibers, Inc. of Mobile, Alabama. Tencel fiber is reported to have high wet and dry tensile strength and modulus in comparison to standard rayon. Table 1 shows Courtauld's reported Tencel fiber properties<sup>3</sup> along with viscose rayon, high wet modulus (HWM) rayon, cotton, polyester and high tenacity nylon. The stress-strain curve of Tencel in comparison to these fibers is provided in Figure 1. Tencel is a premium-priced fiber, and based on calendar year 2000 quotes, Tencel sells for \$1.45 per pound (1.5 denier per filament (dpf)) versus \$2.13 per pound for Type 420 nylon (1.7dpf), approximately \$0.60 per pound for cotton, and \$0.60 per pound for commodity polyester (1.5 dpf).

Tencel is known to fibrillate, which results in surface fuzz on the finished fabric.<sup>2</sup> Fibrillation is an undesirable characteristic. However, some believe it may enhance fabric hand and bulk. Lenzing AG is developing a new non-fibrillating lyocell fiber<sup>2</sup>, but, it was not available for inclusion in this evaluation.

TABLE 1. Fiber Properties

Fiber Property	Tencel	Viscose Rayon	HWM Rayon	Cotton*	Type 420 Nylon	Polyester
Denier per Filament (dpf)	1.5	1.5	1.5	_	1.7	1.5
Tenacity (g/denier)	4.8 - 5.0	2.6 - 3.1	4.1 - 4.3	2.4 – 2.9	6.9	4.8 - 6.0
Elongation (%)	14 - 16	20 – 25	13 – 14	7 - 9	46	44 - 45
Wet Tenacity (g/denier)	4.2 - 4.6	1.2 - 1.8	2.3 - 2.5	3.1 – 3.6	-	4. 8 - 6.0
Specific Gravity	1.50	1.50	1.50	1.50	1.14	1.38
Moisture Regain, % @70 <sup>0</sup> , 65% R.H.	11.5	-	-	8.5	4.0 - 4.5	0.4
Wet Elongation, %	16 - 18	25 – 30	13 – 15	12 – 14	_	44 - 45
Water Inhibition (%)**	65	90	75	50	-	3

<sup>\*</sup> U.S. Middling

<sup>\*\*</sup> A measure of the water holding capacity of a textile fiber

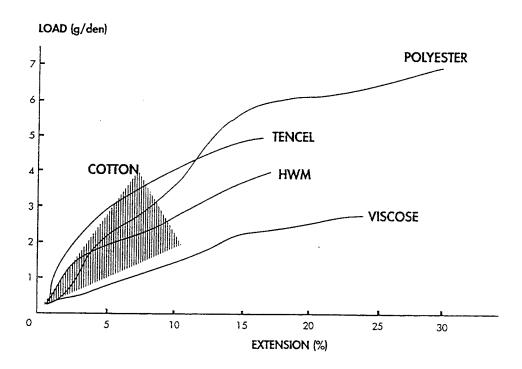


Figure 1. Stress-Strain Curve Comparison

#### **FABRIC DESCRIPTION**

The four fabrics evaluated in this effort are identified as follows:

- a) VEE 6695A (DPT Tencel) 100 percent Tencel, 6.6 ounces per square yard, ripstop poplin, woodland camouflage pattern, manufactured in accordance with MIL-C-43468, Cloth, Camouflage Pattern, Wind Resistant Poplin, Cotton, except that Tencel was used rather than the specified cotton. The fabric was manufactured by Reeves Brothers, Inc., New York, printed at Bradford Dyeing Association, Rhode Island and finished with a Reeveset LFR® durable press treatment at Reeves' Bishopsville Finishing Division.
- b) VEE 6695B (FRT Tencel) 100 percent Tencel, 7.4 ounces per square yard, ripstop poplin, woodland camouflage pattern, manufactured as described above, except that instead of being finished with a durable press treatment it was given an ammonia cure Proban<sup>®</sup> flame retardant treatment at Westex, Inc.
- c) VEE 6561 100 percent cotton, 6.0 ounces per square yard, ripstop poplin, manufactured in accordance with MIL-C-43468. This fabric, obtained from Defense Supply Center Philadelphia, was formerly the standard fabric used in Hot Weather Battledress Uniform.
- d) VEE 6562 6.3 ounces per square yard, ripstop poplin, 50 percent Type 420 nylon (1. 7 dpf), 50 percent combed cotton manufactured in accordance with MIL-C-44436, Cloth, Camouflage Pattern, Wind Resistant Poplin, Nylon/Cotton Blend. The fabric was woven by Marion Mills, printed and finished by Bradford Dyeing Association and purchased under Natick Contract No. DAAK60-90-C-0080. This fabric is the current standard used in the Hot Weather Battledress Uniform.

## **TEST METHODOLOGY**

All tests were performed in accordance with American Society for Testing and Materials (ASTM) and American Association of Textile Chemists and Colorists (AATCC) methods shown in the Appendix, with the exception of the spectral reflectance, which was tested in accordance with the identified specification. Three types of abrasion tests were employed. The abradant used in ASTM-D-4157 was an oscillating cylinder covered with 320 J grade sand paper. ASTM-D-3884 used an H-18 Calibrade wheel. The end point of both ASTM-D-4157 and ASTM-D-3884 abrasion tests was measured by breakage of both a warp and a filling yarn at the same cross over point to form a hole. Both tests and the types of abradants used are intended to simulate hard surface abrasion that may be encountered when kneeling on gritty dirt or sand. The abradant in ASTM-D-3885 was a smooth carbide tipped steel bar, and the end point was the complete rupture of the sample. This test is intended to simulate the abrasion that may occur at the edges of collars and cuffs.

#### **RESULTS AND DISCUSSIONS**

Physical property data of the four fabrics tested are listed in Table 2. The DPT Tencel, cotton, and nylon/cotton fabrics are similarly constructed, and all, except for the FRT Tencel, are within the desired weight range of 6.0 - 7.0 ounces per square yard. The DPT Tencel fabric has the highest air permeability of 27 cubic feet per minute per square foot, and the nylon/cotton fabric has the lowest at 7 cubic feet per minute per square foot.

The difference is due to the low specific gravity and high bulk of the nylon fiber, which provides greater fabric cover than the Tencel and cotton fibers.

TABLE 2. Physical Properties of Tencel, Cotton, and Nylon/Cotton Ripstop Poplin Woodland Camouflage Fabrics

Characteristic	VEE 6695A 100% Tencel DPT	VEE 6695B 100% Tencel Proban FRT	VEE 6561 MIL-C-43468 100% Cotton	VEE 6562 MIL-C-44436 50% Nylon, 50%Cotton
Weight, oz/yd <sup>2</sup>	6.6	7.4	6	6.3
Thickness, In.	0.015	0.015	0.015	0.016
Yarn Count/Ply Warp x Filling	37/2 x 16/1	37/2 x 16/1	40/2 x 17/1	40/2 x 18/1
Fabric Count Warp x Filling	104 x 53	104 x 52	107 x 52	104 x 55
Air Permeability, Ft <sup>3</sup> /min/ft <sup>2</sup>	27	18	13	7
Tearing Strength, lbs Warp x Filling Initial Wet Washed 5X	12.3 x 21.6 9.5 x 7.5 15.3 x 23.6	6.2 x 7.0 - -	4.0 x 5.1 - 3.4 x 3.9	8.6 x 5.9 - 9.5 x 4.8
Breaking Strength, lbs. Warp x Filling	205 x 152	180 x 117	165 x 79	264 x 107
Elongation, % Warp x Filling	14 x 10	11 x 8	8 x 8	37 x 19
Abrasion Resistance, Cycles After 5 Wash Cycles ASTM D-3884 ASTM D-4157 Warp x Filling ASTM D-3885 Warp x Filling	164 42 x 41 449 x 494	-	229 90 x 60 450 x 425	804 170 x 275 4,550 x 7,000
Dimensional Stability, % Warp x Filling	1.4 x 0.4	-	-	-

The DPT Tencel fabric demonstrates unusually good tearing strength with  $12.3 \, \mathrm{x}$  21.6 pounds (warp x filling) compared to the cotton and nylon/cotton fabrics with  $4.0 \, \mathrm{x}$  5.1 and  $8.6 \, \mathrm{x}$  5.9 pounds, respectively. The increased tearing strength may be attributed to both the coarser yarns used, and the slightly more open structure of the Tencel fabric which allows yarn grouping to share the tensile load. Although the reported Tencel fiber tenacity drops slightly when wet (see Table 1), the fabric when tested wet, lost 22 percent of its strength in the warp direction, and 65 percent in the filling direction. While this loss is substantial, the wet tearing strength of  $9.5 \, \mathrm{x}$  7.5 pounds is comparable to the dry nylon/cotton fabric both initially ( $8.6 \, \mathrm{x}$  5.9 pounds) and after five washings ( $9.5 \, \mathrm{x}$  4.8 pounds) and therefore is considered acceptable.

The nylon/cotton fabric demonstrates the highest warp breaking strength of 264 pounds due to the high tenacity nylon (6.9 g/denier vs. Tencel at 5.0) followed by the Tencel fabric with 205 pounds, and the cotton fabric with 165 pounds. In the filling direction, the Tencel fabric has the highest strength with 152 pounds, followed by the nylon/cotton with 107 pounds, and the cotton with 79 pounds.

All three fabrics were evaluated for abrasion resistance after five wash cycles (washing method in accordance with AATCC-96). The nylon/cotton fabric demonstrates the greatest abrasion resistance. Since all three fabrics are constructed similarly, the greater abrasion resistance of the nylon/cotton fabric is attributed to the higher elongation and energy absorbing properties of the nylon fiber. There is little difference in the abrasion resistance of the DPT Tencel and the cotton fabric because both are cellulosic based.

The DPT Tencel fabric does not meet the target performance goals for visual shade match, near infrared spectral reflectance, and colorfastness to crocking and laundering, when compared to MIL-C-43468, as shown in Tables 3 and 4. It was compared to MIL-C-43468 because they are both cellulosic based fabrics and would be expected to perform similarly. The near infrared spectral reflectance was also compared to MIL-C-44031 because it is the Master Standard, but failed to meet these requirements as expected. See Table 5. While it is expected that the dye and print formulations can be adjusted to improve the visual, near infrared, and colorfastness properties to the level of MIL-C-43468, it is unlikely they will meet the performance of the Master Standard, which is based on the performance of acid dyes. Acid dyes are only compatible with nylon fiber.

The FRT Tencel fabric at 7.4 ounces per square yard exceeds the target weight range of 6.0 to 7.0 ounces per square yard. The application of the flame retardant treatment adds approximately 20 percent to the finished fabric's weight. The additional weight add-on may have caused fiber and yarn swelling, decreasing yarn mobility which may account for the substantial reduction in tearing strength of 50 and 68 percent (warp x filling). Flame retardant treatments are also known to tender cellulosic fabrics. The warp and filling breaking strengths also decreased, but not as dramatically as the tearing strength, by 12 and 23 percent, respectively. In addition, the FRT Tencel fabric demonstrated substantially lower tearing strength (6.2 x 7.0) than the DPT Tencel, however, the FRT Tencel fabric was comparable to the nylon/cotton fabric and therefore is acceptable. Abrasion testing was not conducted on the FRT Tencel because it would not be expected to be greater than the DPT Tencel. As shown in Table 6, the fabric demonstrates good flame resistance.

TABLE 3. Visual and Colorfastness Properties of Tencel DPT Ripstop Poplin Fabric Compared to MIL-C-43468, 100 Percent Cotton

Fiber Properties	Light Green 354	Dark Green 355	Dark Brown 356	Black 357
Visual Shade 1/	Fail (too full & green)	Fail (too full & blue)	Fail (too yellow)	Fail (too thick & blue)
Colorfastness to: Light: 40 SFH 2/	Good	Excellent	Excellent	Excellent
Crocking: Dry Wet	3.5 2.5 3/	3.0 3/ 2.0 3/	3.0 3/ 1.5 3/	3.0 1.0
Perspiration: Alkaline Acid	Excellent Excellent	Excellent Excellent 4/	Excellent Excellent	Excellent Excellent 4/
Laundering: Sample Cotton Acetate Polyester Acrylic Wool Nylon	Fair Excellent Good Good Good Good Fair	Fair Excellent Good Good Fair Good Fair	Poor Excellent Good Good Fair Good Fair	Fair Poor Fair Fair Foor Poor

<sup>1/</sup> In comparison to Standard Roll #3347.

<sup>2/</sup> Standard fading hours (SFH).

<sup>3/</sup> Does not meet performance goal - AATCC Chromatic Scale Rating of not less than 3.5 or ≥ standard sample.

<sup>4/</sup> Exception: nylon fiber – good.

TABLE 4. Spectral Reflectance in the Near-Infrared of Tencel DPT Fabric Compared to MIL-C-43468, 100 Percent Cotton

Light Gr	een 354	Į	Dark G	reen 355	and Dark B	rown 356	Black 3	357
Target		Tencel	Target		Tencel		Target	Tencel
Max	Min_		Max	Min	Green	Brown	Max	
21	8	11	10	3	4	5	10	2
21	8	10	10	3	4	5	10	2
21	8	10	10	3	4	5		3
22	8	10	12	3	4	5		3
27	10	11	16	3	4	5		3
40	13	*12	18	4	5	6		4
53	16	*15	20	5	6	7		4
64	21	*19	28	7	8	9		4
73	27	*24	36	11	11	13		5
80	34	*31	44	17	*16			5
85	41	*38	52	24	*23			5
88	48	*43	60	32				6
90	50	*48	68	39				6
91	52	52	74	46	*41	*40	10	7
	Target  Max  21  21  22  27  40  53  64  73  80  85  88  90	Target  Max Min  21 8  21 8  21 8  21 8  22 8  27 10  40 13  53 16  64 21  73 27  80 34  85 41  88 48  90 50	Max Min  21 8 11  21 8 10  21 8 10  21 8 10  22 8 10  27 10 11  40 13 *12  53 16 *15  64 21 *19  73 27 *24  80 34 *31  85 41 *38  88 48 *43  90 50 *48	Target         Tencel         Target           Max         Min         Max           21         8         11         10           21         8         10         10           21         8         10         10           22         8         10         12           27         10         11         16           40         13         *12         18           53         16         *15         20           64         21         *19         28           73         27         *24         36           80         34         *31         44           85         41         *38         52           88         48         *43         60           90         50         *48         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<sup>\*</sup> Failed to meet target range.

TABLE 5. Spectral Reflectance in the Near-Infrared of Tencel DPT Fabric Compared to MIL-C-44031, Twill (Master Standard)

	_		n_354	Dark Gr	een 355 :	and Dark Br	own_356	Black 3	57
Wavelength	Targe	t	Tencel	Target		Tencel		Target	Tencel
	Max	Min		Max	Min	Green	Brown	_Max	
600	18	8	11	10	3	4	5	10	2
620	18	8	10	10	3	4	5	10	2
640	18	8	10	9	3	4	5	10	3
660	18	8	10	12	3	4	5	10	3
680	22	10	11	14	3	4	5	10	3
700	33	18	*12	18	5	5	6	10	4
720	45	22	*15	20	7	*6	7	10	4
740	55	30	*19	28	12	*8	*9	10	4
760	65	35	*24	36	18	*11	*13	10	5
780	75	40	*31	44	26	*16	*18	10	5
800	80	45	*38	52	34	*23	*24	10	5
820	86	50	*43	60	42	*30	*30	10	6
840	88	55	*48	68	50	*36	*35	10	6
860	90	60	*52_	74	56	*41	*40	10	7

<sup>\*</sup> Failed to meet target range.

TABLE 6. Flame Resistance Properties of Proban® FR Treated Tencel Fabric

Properties	VEE 6695B 100% Tencel Proban FRT	Performance Goals
After Flame, seconds Warp x Filling	0 x 0	2.0 x 2.0 (max.)
After Glow, seconds Warp x Filling	3.5 x 2	25.0 x 25.0 (max.)
Char Length, inch Warp x Filling	3.6 x 3.4	4.0 x 4.0 (max.)

In regards to fibrillation, the Tencel fabrics did not exhibit any surface fuzz upon receipt, but did exhibit it after laundering. In addition to surface fuzz the fabrics also demonstrate surface frosting. While subjective, the surface fuzz and frosted appearance are considered to be objectionable by the author.

The feasibility of spinning Tencel and nylon blend yarn was examined in a follow-on effort. DuPont ran yarn spinning trials on 50 percent Type 420 nylon (1.7 dpf) and 50 percent Tencel (1.5 dpf) and spun two yarn sizes, i.e. 40/1 and 19/1cotton count. The yarns were run on commercial equipment and compared with yarns of Type 420 nylon and combed cotton. The yarn spinning trials were conducted without incident. Results of the spinning trials provided by Dupont are listed in Table 7. The nylon/Tencel blend demonstrates the following advantages over the nylon/cotton blend: better along-end uniformity of sliver, roving, and yarn; improved yarn quality (fewer defects), and much higher yarn breaking strength.

TABLE 7. Sliver and Spun Yarn Properties of Nylon 420/Tencel Vs Nylon 420/Cotton Blends

Yarn Properties	l l	Nylon/Combed Cotton Draw Frame Blend	50/50 Nylon/Tencel Intimate Blend		
Uniformity, % CV* Card Sliver Draw I Sliver Draw II Sliver Roving	(9.6)** 4.1 3.0 5.4		6.3 3.5 2.9 4.7		
Yarn Quality % CV Thick Thin Neps  Yarn Break Factor, ASTM-	40/1 17.1 414 73 268 2580	_19/1 12.0 66 0 43	40/1 15.6 46 29 13		

<sup>\*</sup> A Uster tester was used to generate this data. It provides a continuous measurement of the variation in weight per unit length of sliver, roving, and yarn.

#### CONCLUSIONS

The DPT Tencel fabric demonstrates greater breaking strength and tearing strength over the former standard cotton ripstop fabric, most likely due to the greater tenacity of the Tencel fiber, and equivalent abrasion resistance. Adjustment to the dye and print formulations for the Tencel fabric will likely improve the visual shade match, near infrared spectral reflectance, and colorfastness to crocking and laundering, to meet the requirements of the former standard MIL-C-43468. The addition of the flame retardant treatment increased the weight of the fabric and decreased its breaking and tearing strengths. The FRT Tencel fabric exhibited good flame resistance. Both the FRT Tencel and DPT Tencel fabrics demonstrated fibrillation and frosting that is considered unacceptable by the author. The DPT Tencel fabric demonstrates greater filling breaking strength, tearing strength, and air permeability than the nylon/cotton ripstop fabric. The nylon/cotton fabric, which is the current Hot Weather Battledress Uniform fabric, has the greatest resistance to abrasion, is known to meet all visual, near-infrared, and colorfastness requirements, and is not known to fibrillate. Spinning of Type 420 nylon and Tencel 50/50 blends is feasible and offers advantages in uniformity, yarn quality and breaking strength over the standard nylon/cotton blend yarn.

<sup>\*\* 100</sup> percent Type 420 Nylon (1.7 dpf) card sliver, blended with combed cotton at Draw Frame.

#### RECOMMENDATIONS

While the Tencel DPT demonstrated improved performance for several characteristics over the standard nylon/cotton blend Hot Weather Battledress Uniform fabric, it fell short in terms of abrasion resistance, visual color, near-infrared spectral reflectance, colorfastness, and appearance (fibrillation). It is not recommended for further investigation. The FRT Tencel is not recommended for further investigation due to increased fabric weight and relatively low breaking and tearing strengths.

Due to the promising results of the yarn spinning trials it is recommended that Tencel fiber be considered as a substitute for cotton in the standard Hot Weather Battledress Uniform fabric. The military user community would need to decide if the potential improvement in durability would substantiate the increased cost of Tencel over cotton. While the fibrillation would be reduced by 50 percent, prototype fabrics would need to be made to assess the subjective acceptability of the surface fuzz and frosting, or a non-fibrillating version of Tencel could be used if commercially available.

This document reports research undertaken at the U.S. Army Soldier and Biological Chemical Command, Soldier Systems Center, Natick, MA, and has been assigned No. NATICK/TR-02/007 in a series of reports approved for publication.

# **REFERENCES**

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- 2. S. G. Maycumber, "Lenzing Developing New Lyocell", <u>Daily News Record</u>, New York, New York, 14 October 1993, p. 8
- 3. Courtaulds Fibers, Tencel® The Nature of Excellence, Courtaulds company literature

# **APPENDIX**

# Test Methods

AATCC Method No. AATCC-8	Title Colorfastness to Crocking: AATCC Crockmeter
AATCC-15	Colorfastness to Perspiration
AATCC-16 (Opt. A)	Colorfastness to Light
AATCC-61	Colorfastness to Laundering, Home and Commercial: Accelerated
AATCC-96 (Test VI, A)	Dimensional Changes in Commercial Laundering of Woven and Knitted Fabrics Except Wool
ASTM Method No. ASTM 737	Title Air Permeability of Textile Fabrics
ASTM D 1424	Tear Resistance of Woven Fabric by Falling Pendulum (Elmendorf) Apparatus
ASTM D 1777	Thickness of Textile Materials
ASTM D 3775	Fabric Count of Woven Fabric
ASTM D 3776	Mass Per Unit Area (Weight) of Fabric Option C
ASTM D 3884	Abrasion Resistance of Textile Fabrics (Rotary Platform, Double-Head Method)
ASTM D 3885	Abrasion Resistance of Textile Fabrics (Flexing and Abrasion Method)
ASTM D 4157	Abrasion Resistance of Textile Fabrics (Oscillatory Cylinder Method)
ASTM D 5034	Standard Test Method for Breaking Strength and Elongation; Grab Test